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METHODS OF CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART II DETERMINATION OF SILVER, COPPER AND TIN

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METHODS OF CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART II DETERMINATION OF SILVER, COPPER AND TIN

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART II DETERMINATION OF SILVER, COPPER AND TIN

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 31 October 1969, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 In IS : 2927-1964*, 24 grades of silver-copper brazing alloys have been specified. In order to determine correctly the composition of different silver-copper brazing alloys, methods of analysis have been described in various parts of this standard. Determination of silver and copper as prescribed for silver-copper brazing alloys except BA-CuAg7 and BA-CuAg8 is covered in IS : 4667 (Part I)-1968. This standard (Part II) describes the methods for silver, copper and tin in BA-CuAg7 and BA-CuAg8. Methods for analysis of zinc, cadmium and nickel will be covered in subsequent parts.

0.3 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the ' 1967 Book of ASTM methods for chemical analysis of metals : Part 32 ' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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1. SCOPE

1.1 This standard (Part II) prescribes methods of chemical analysis for tin, silver and copper in alloys BA-CuAg7 and BA-CuAg8 as specified in IS : 2927-1964*.

*Specification for brazing alloys.

†Rules for rounding off numerical values (*revised*).

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS : 1817-1961*.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in the tests.

NOTE — ' Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF TIN BY THE GRAVIMETRIC METHOD

4.1 Outline of Method — Tin is precipitated as metastannic acid and is ignited and volatilized with ammonium iodide. The loss in weight represents tin oxide.

4.2 Reagents

4.2.1 *Dilute Nitric Acid* — 1 : 1 and 3 : 97 (*v/v*).

4.2.2 *Ammonium Iodide* — solid.

4.2.3 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961‡).

4.3 Procedure

4.3.1 Transfer 1 g of an accurately weighed sample into a 250-ml beaker. Add 20 ml of dilute nitric acid (1 : 1) and place on a hot plate. Evaporate to syrupy consistency, add 100 ml of hot water and 10 ml of dilute nitric acid (1 : 1). Place on the hot plate for one hour, filter the precipitated metastannic acid, wash with dilute nitric acid (3 : 97) and preserve the filtrate.

4.3.2 Dry the precipitate in the oven and transfer the filter paper containing tin oxide residue to a weighed porcelain crucible. Ignite and heat it in a muffle furnace at 900°C to constant weight.

4.3.3 Heat to volatilize tin with 5 g of ammonium iodide. Treat the residue with a drop of concentrated sulphuric acid. Heat to fumes, ignite and weigh. Loss in weight represents tin oxide. Take up the residue after volatilization with 10 ml of dilute nitric acid (1 : 1) and add to the filtrate preserved under **4.3.1.** Transfer to a 250-ml flask and dilute to the mark. Reserve the solution for further determinations.

*Methods of sampling non-ferrous metals for chemical analysis.

†Specification for water, distilled quality (*revised*).

‡Specification for sulphuric acid (*revised*).

4.4 Calculation

$$\text{Tin, percent} = \frac{A \times 78{\cdot}76}{B}$$

where

A = loss in weight in g due to volatilization of tin oxide, and

B = weight in g of the sample taken.

5. DETERMINATION OF SILVER BY THE GRAVIMETRIC METHOD

5.1 Outline of Method — After separation of tin from the solution of the sample, silver is precipitated as silver chloride by means of hydrochloric acid. The precipitate is filtered, washed, dried and weighed.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 3 : 97 (*v*/*v*).

5.2.2 *Dilute Hydrochloric Acid* — 1 : 19 (*v*/*v*).

5.3 Procedure

5.3.1 Take a suitable aliquot from the solution preserved under **4.3.3** and boil. Add sufficient amount of dilute hydrochloric acid slowly with constant stirring till the precipitation is complete. Add a few ml of dilute hydrochloric acid in excess. Let it stand for about an hour, keeping it warm.

5.3.2 Decant the solution through a weighed sintered glass crucible. Wash the precipitate twice with warm dilute nitric acid and decant through the crucible. Transfer the precipitate to the crucible and wash with hot water till free from chloride. Transfer the filtrate and the washings to a 400-ml beaker and reserve for the determination of copper (*see* **6.4.1**).

5.3.3 Dry the crucible and precipitate at 120° to 130°C for 1 to 1½ hours. Cool in a desiccator to room temperature and weigh.

5.4 Calculation

$$\text{Silver, percent} = \frac{A}{B} \times 75{\cdot}26$$

where

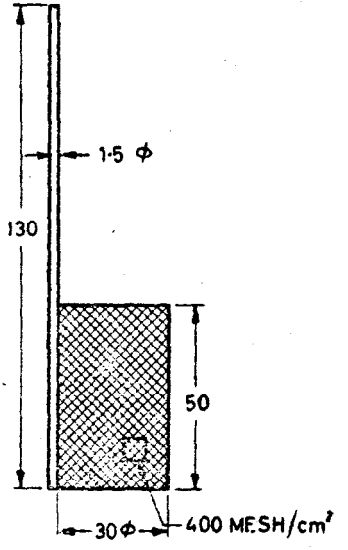
A = weight in g of silver chloride, and

B = weight in g of the sample represented by the aliquot taken.

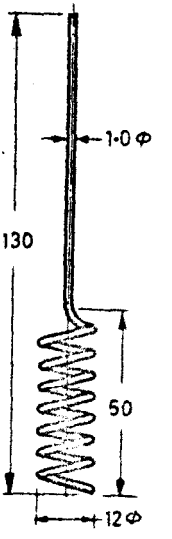
6. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

6.1 Outline of Method — After the separation of tin and silver from the solution of the sample, copper is deposited electrolytically from the solution and weighed.

6.2 Apparatus — The following platinum electrodes (*see* Fig. 1) are recommended but strict adherence to the shape and size of the electrodes is not essential. In order to decrease the time of the deposition, one of the types of rotating forms of electrodes generally available for agitation of the electrolyte may be employed.



1A Cylindrical Platinum Cathode



1B Spiral Platinum Anode

All dimensions in millimetres.

FIG. 1 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

6.2.1 *Cathode* — It may be formed either from plain or perforated sheet or from wire gauze.

6.2.1.1 Gauge cathodes made preferably from gauze containing 400-mesh/cm² should be used. The wire used for making gauze should be approximately 0·20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about 3 mm on the top and bottom or by reinforcing the gauze at the top and bottom or with a platinum ring or band.

6.2.1.2 The diameter and height of the cylinder should be approximately 30 mm and 50 mm respectively. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1·5 mm. It should

be flattened and welded to the entire height of the gauze. The overall height of the cathode including the stem should approximately be 130 mm.

6.2.2 *Anode* — Either a spiral or a gauze anode should be used. The spiral anode should be made from 1·0 mm or larger platinum wire formed into a spiral of seven coils with a height of approximately 50 mm and diameter of 12 mm, the overall height including the stem being 130 mm. The gauze anode should be made of the same material and of the same general design as platinum gauze cathode specified under **6.2.1.**

6.3 Reagents

6.3.1 *Concentrated Sulphuric Acid — see* **4.2.3.**

6.3.2 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1968*).

6.3.3 *Urea* — solid.

6.3.4 *Ethanol or Methanol* — 95 percent (*v/v*).

6.4 Procedure

6.4.1 To the filtrate and washings reserved under **5.3.2**, add 5 ml of concentrated sulphuric acid and evaporate to fumes, cool, dilute to 200 ml and add 5 ml of concentrated nitric acid. Add 2 g of urea, insert the electrodes, the cathode having been accurately weighed; cover with a pair of split cover-glass and electrolyze for 16 hours at a current density of 0·6 A/dm^2 (at this current density, the electrolysis is conveniently carried on overnight), or at a current density of 4 A/dm^2 for a short period (about 2 hours). In the latter case, one of the types of rotating forms of electrodes generally available may be used. When the solution becomes colourless reduce the current density to 0·3 A/dm^2 and continue electrolysis until the deposition of copper is complete as indicated by the absence of plating on the new surface of the electrode obtained by lowering the electrode.

6.4.2 Without interrupting the current, lower the beaker slowly while washing thoroughly with water and collecting the washings into the electrolyte. Remove the cathode quickly while washing further with water; rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air oven at 100°C for three to five minutes, cool and weigh for copper.

6.5 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper, and

B = weight in g of the sample represented by the aliquot taken.

*Specification for nitric acid (*first revision*).

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